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Effect of compaction water content on the strength of cement-stabilised rammed earth materials

Christopher Beckett¹ and Daniela Ciano

Abstract:

Current guidelines suggest that stabilised rammed earth materials be compacted at their optimum water content in order to achieve their maximum strength. Although this is true for traditional rammed earth, there is no evidence that this procedure should also be used for cement-stabilised rammed earth. Furthermore, the water content used at compaction is usually difficult to control on a construction site, so that material might be compacted at water contents other than the optimum. In this paper, a novel experimental programme is presented in which the effect of compaction water content on the unconfined compressive strength of crushed limestone stabilised to 5% Portland cement content is investigated for a range of curing periods. Freeze drying of specimens was used to arrest cement hydration in order to determine the evolution of hydrated cement content. SEM analysis was used to identify differences between the final material microstructures. Results are discussed demonstrating the intimate link between the amount of hydrated cement, material microstructure and compressive strength.

Key words: Rammed earth; compaction; freeze drying; cement hydration.

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1. Introduction

It is well understood that strength of dry, unstabilised rammed earth (URE) materials is related to the level of suction developed between the soil particles and the material's dry density (Jaquin et al., 2009). For a given compactive effort, higher strengths can therefore be achieved by compacting the raw material at its Optimum Water Content (OWC) to ensure that the maximum dry density is achieved.

The RE construction method of the compaction of moist soil into removable formwork has changed little since its conception, however it is now common to stabilise the raw material with small quantities of Portland cement (or some similar stabiliser), in order to improve material strength and durability. Although the sources of strength in stabilised RE (SRE) materials are less clear, the same practices used for URE are applied to SRE construction, in that materials are compacted at their OWC in order to achieve the highest dry density and so, ostensibly, strength. However, the complex interactions between the stabilising agents and water makes the relationship between water content, dry density and strength much harder to predict.

Laboratory determination of the compaction OWC is generally carried out using either the Standard or the Modified Proctor Tests (MPT) (for example as described in AS1289.5.2.1 in Australia, with similar descriptions in other standards around the world). These tests are well specified, codified and widely established and hence are repeatable in any laboratory. Determination of the OWC on site is, however, more complicated due to the use of large quantities of material of unknown and often varying saturation. The "drop test" is a technique recommended by RE guidelines (e.g. Middleton and Schneider (1992); Walker and Standards Australia (2002)) which can be used to approximately identify the OWC under these conditions. Unfortunately, this test is highly subjective on the skills of the operator and can result in a wide range of estimates for the material OWC, with unknown consequences on achieved material strength (Smith and Augarde, 2013). Clearly, this is of supreme importance when designing for material safety.

This paper aims to address this uncertainty by determining the effect of compaction at water contents above, below and at optimum on the Unconfined Compressive Strength (UCS) of a given RE material, stabilised using a set proportion of Portland cement, by means of a detailed laboratory experimental programme. Testing is conducted under laboratory conditions in order to avoid inconsistencies in material preparation inherent in site practice. This programme is described in Section 2, with find-

ings discussed in Section 3. Conclusions arising from this work are then presented in Section 4.

2. Experimental Procedure

2.1. Material

RE construction in Western Australia (WA) generally utilises crushed limestone in the place of natural soil due to improved quality control, aesthetic appeal and ready availability. Crushed limestone has several advantages over the use of natural soil, for example a greater consistency in mineralogy and a low fine particle (especially clay) content, which might otherwise interfere with cement hydration (Croft, 1967; Fernandes et al., 2007). Crushed limestone is therefore used in this study to ensure greater repeatability of results and applicability to the wider area of construction utilising compacted stabilised soils. The crushed limestone particle size distribution as shown in Figure 1. Oven-dried (105°C for 24 hours) crushed limestone was mixed with 5% Portland cement (by mass of dry limestone) for stabilisation. Again, this is typical of WA RE construction, however it is acknowledged that higher Portland cement contents are also used. Crushed limestone used here was sieved to pass 5mm; this is not representative of construction on-site, where aggregates of up to 20mm in size might be encountered (Walker and Standards Australia, 2002), but was necessary to improve quality control and to prevent larger particles from interfering either with the compaction process or in the subsequent image analysis (discussed below).

2.2. Determination of OWC

The Modified Proctor Test (MPT) was used to determine the OWC of the stabilised crushed limestone, in accordance with AS 1289.5.2.1.-2003 (Standards Australia, 2003). Dry crushed limestone was combined with 5% Portland cement by mass and mixed thoroughly for a minimum of five minutes. A known amount of water $w\%$ was then added and the material mixed for a further five minutes. Testing was completed within 45 minutes of wetting in order to prevent cement hydration from interfering with the compaction process. Unlike as specified in AS 1289.5.2.1.-2003, oven drying could not then be used to verify material water content due to the triggering of cement hydration at high temperatures (Korpa and Trettin, 2006; Zhang and Scherer, 2011). Material water content was therefore taken to be equal to $w\%$ for OWC determination. Material OWC and corresponding dry density, ρ_d , are shown in Figure 2. Note that ρ_d values given in Figure 2 correspond to those at compaction and so do not take

74 into account changes in density due to cement hydration.

75 **2.3. Preparation of samples for UCS**

76 In typical design practice and standards (for example Walker and Standards Australia (2002)), UCS
77 is the primary engineering measure of material properties for RE construction. Material strengths deter-
78 mined via UCS testing are therefore used here to compare the effects of compaction at water contents
79 below, at and above optimum, following the work of previous authors (Burroughs, 2008; Jayasinghe
80 et al., 2007; Middleton and Schneider, 1992). Specimens of 100mm diameter, 200mm height were
81 manufactured at water contents of 10.4, 12.4 and 14.4% (OWC−2%, OWC and OWC+2%, hereafter
82 referred to as “L−”, “O−” and “H-series” specimens respectively, as indicated in Figure 2) using the
83 same mixing procedures as discussed above. This range of water contents was selected to be represen-
84 tative of those likely to be encountered in site practice (specimen preparation and handling becomes
85 very difficult outside of this range). Specimens for each series were compacted to the corresponding
86 dry density for that water content, as shown in Figure 2, by compacting a given mass of material to a
87 set volume using a jack hammer and a volume-controlled rammer head as discussed in Beckett (2011).

88 Specimens were wrapped with plastic film immediately following extraction in order to prevent
89 water loss or gain. This is discussed in more detail below and in Section 3. Once wrapped, specimens
90 were cured under conditions of $94 \pm 2\%$ relative humidity and $21 \pm 1^\circ\text{C}$. Curing periods of 1, 3, 5,
91 7, 14 and 28/29 days (depending on equipment availability) were used to monitor the development of
92 strength with curing time. UCS was then determined by crushing specimens uniaxially at a constant
93 displacement rate of 0.3mm/min until failure. By placing Teflon sheets between the specimen and
94 the loading platens, the effects of end friction could be negated and the UCS determined directly by
95 dividing the peak sustained load by the nominal specimen cross sectional area without the use of
96 slenderness factors (Ciancio and Gibbings, 2012). Crushed material was then oven dried at 105°C for
97 24 hours in order to determine material water content. Three specimens were tested per series and
98 curing time (i.e. 56 in total).

99 **2.4. Further tests: SEM and FD**

100 Small, intact fragments of nominal size 4mm were taken from 28-day cured crushed material (all
101 series) for Scanning Electron Microscope (SEM) analysis. Three techniques were investigated to de-

hydrate samples in order to arrest cement hydration: ethanol-water wash (six individual washes of concentrations 30%, 50%, 90% and three successive washes at 100% ethanol to remove water from the pore space) followed by critical point drying; freeze drying/sublimation at -80°C ; and freeze drying/sublimation at -30°C . It was found that the combined ethanol wash and critical point drying technique (EW/CP) resulted in the least damage to the material microstructure, as determined from SEM images of treated fragments. A second round of 28-day cured fragments (nominal size 4mm) was therefore prepared using the EW/CP method. Fragments were then coated with a 6nm thick layer of platinum and stored in a desiccator until needed.

In addition to UCS testing, one specimen per series and curing time was prepared for freeze drying (hereafter referred to as “FD” specimens) in order to determine the evolution and extent of cement hydration. Cylindrical specimens of 100mm diameter and 80mm height were manufactured following the same mixing, compaction and wrapping/curing procedures discussed above. Freeze drying is used here as, unlike oven drying, it arrests cement hydration and maintains original material structure without the need to immerse or wash specimens, which would cause damage to immature material. The mass lost on drying therefore equals the amount of free water (i.e. liquid water residing in the material pore spaces) present in the material (Zhang and Scherer, 2011). As specimens were wrapped, the amount of water used for cement hydration can then be determined as the difference between the initial and free water contents. Furthermore, as curing conditions for UCS and FD specimens were identical, the water content obtained via freeze drying can be directly compared to that measured via oven drying, in order to identify differences between the two techniques; the results of this comparison are discussed in the following section.

3. Results and Discussion

Results for specimen UCS with curing time are shown in Figure 3 for all series. Trends of series averages are also shown. Bryan (1988a) presented a series of studies wherein the strengths of Portland-cement stabilised soils were related to their dry density at testing. In those works, a range of soil types (with a maximum clay content of roughly 52% by mass) from the South West of England were stabilised by adding 7.5% Portland cement content by mass and compacted at their OWCs under a static load of 2 MPa to their maximum dry density. Results indicated that those soils with higher initial maximum dry densities (i.e. prior to cement hydration) also displayed higher strengths. However, no

data is available to determine which of the tested textures achieved these higher dry densities, so that comparisons are difficult to substantiate. A similar result is presented in Ciancio et al. (2012) for five engineered soil mixes comprising different combinations and quantities of Portland cement or hydrated lime (maximum combined content 6% by mass), compacted using a controlled compactive energy at the individual material OWC: the higher the specimen dry density at testing, the higher the strength.

Contrary to unstabilised rammed earth, where there is nominally no difference between dry density at compaction and at testing, such a difference exists for cement-stabilised rammed earth due to the hydration of cement (the dry density at testing is expected to be higher than that at compaction). The average dry density at 28 or 29 days is reported in Figure 3 for each tested series. Results reported in the same Figure show that L-series specimens, manufactured to the lowest initial value of ρ_d and showing the lowest value of ρ_d at time of testing, achieved higher UCS values, with a higher rate of strength gain than those of series O and H for all tested curing periods. Although apparently contradictory to the studies discussed above, it must be noted that changes in ρ_d shown in Figure 2 are due to compaction of a given material at water contents above or below its optimum for the same compactive effort, as opposed to compaction at optimum for different materials. These results therefore highlight the importance of consideration of the compaction conditions when interpreting density-versus-strength correlations. Despite this, results shown in Figure 3 suggest that lower compaction water contents produce higher values of UCS. This result recalls the well understood practise in concrete where lower water-cement ratios (w/c) are preferred to achieve higher strengths. However, it also contradicts the traditional association of maximum strengths with maximum dry density ($\rho_{d,max}$) at compaction and so deserves closer inspection.

Given that specimens were wrapped during curing, the amount of water lost to cement hydration (the “hydration water”) can be determined as the difference between the specimen’s initial water content and the amount of free (i.e. unbound) water present in the material. The mass of hydration water is proportionate to the mass of hydrated cement and so can be used as a direct measure of the amount of hydrated cement present in the material at a given time. Freeze drying was used to halt cement hydration after different curing periods, in order to determine hydration water contents. Results for freeze drying tests are given in Table 1.

Freeze drying results were compared to oven drying results (post UCS testing) in order to investigate the abilities of each method to determine material water contents. Figure 4 shows that freeze

161 drying is more sensitive to changes in water content with curing time and is able to detect higher wa-
162 ter contents than oven drying, particularly for shorter curing periods. This is most evident for tests
163 conducted on H-series material, with recorded differences between freeze drying and oven drying of
164 1.3% at 1 day, reducing to $<0.1\%$ at 29 days, but for which oven drying detects little-to-no change in
165 water content over the same period. This lack of sensitivity is suggestibly due to the acceleration of the
166 cement hydration process on exposure to higher temperatures, which results in some of the free water
167 present being lost to cement hydration, as opposed to evaporation. As it arrests cement hydration, this
168 is not a concern for material dried using the freeze drying technique. However, although detected dif-
169 ferences are dependent on the amount of cement present (5% in this work), results shown in Figure 4
170 suggest that oven drying might be acceptable for determining the water contents of mature (i.e. curing
171 periods of >28 days) material for which the majority of cement is hydrated.

172 A comparison between the hydration water and specimen UCS for each series is shown in Figure 5;
173 such a comparison is valid due to the identical manufacturing and curing conditions used for UCS and
174 FD specimens. Given that water could not be gained or lost during curing, due to the use of wrapping,
175 the rate of increase in hydration water directly reflects the rate of cement hydration. Hydration water
176 quantities can be used to determine relative hydrated cement contents, e.g. 40.7% and 54.1% more
177 cement hydrated in O- and H-series specimens than in L-series specimens respectively for the longest
178 curing times.

179 Given that series initial cement contents were very similar (between 58.4 and 59.5g depending
180 on specimen density), it is clear from Figure 5 that not all cement present in each of the materials
181 was able to hydrate. Figure 5 shows that higher rates of hydration and final hydrated cement contents
182 were achieved by materials prepared at higher compaction water contents. However, series gain in
183 strength through time, as shown in Figure 3, appears to be indirectly proportional to the increase in the
184 amount of hydrated cement; whereas L-series specimens lost around 17g of water to hydration (and
185 so developed a proportional amount of hydrated cement) over 28 days, to achieve a final strength of
186 almost 5 MPa, H-series specimens lost around 26g of water over the same period (representing a larger
187 amount of hydrated cement than present in the L-series), for a final strength of only around 2 MPa.
188 This result seems highly counter-intuitive, as it would be expected that the material comprising the
189 largest amount of hydrated cement would, in turn, display the highest values of UCS. SEM analyses,
190 as discussed in Section 2, were therefore conducted on intact mature material fragments in order to

investigate properties of their microstructures and how they might affect the strength of the material.

Micrographs of fragments of L-, O- and H-series material are shown in Figures 6 to 8 respectively.

Figure 6 shows that L-series material is characterised by sub-angular limestone particles/aggregates (between 100 and 500 μm), with the majority of the cement found in the form of smooth hydrated cement bridges (highlighted, Figure 6(a)). This aggregated/bridged structure gives rise to the formation of large (up to 200 μm) interaggregate voids.

Figure 7 shows that large (up to 500 μm) voids are still present in O-series material. Some larger limestone particles/aggregates (between 100 and 500 μm) are seen in isolation but the large majority are surrounded by a fine particle matrix. Regions of smooth hydrated cement are seen both covering the surface of this fine matrix (Figures 7(b) and (c)) and in the form of cement bridges, as present in L-series material. Void interconnectivity is difficult to comment on, given the lack of internal information, however it is likely that voids present in O-series material are less interconnected than those seen in Figure 6 for L-series material due to the presence of the fine matrix (Beckett et al., 2013).

Figure 8 shows that larger limestone particles (between 100 and 500 μm) are no longer found either in isolation or in the form of aggregates in H-series material but instead are inundated by the fine particle matrix. As such, no large voids, as visible in Figures 6 and 7, are present. Distinct regions of hydrated cement are difficult to identify, suggesting that cement is instead entirely held within the matrix.

The development of the fine particle matrix, and the consequent loss of distinct regions of hydrated cement, as compaction water contents increase from L- to H-series offers an explanation for the apparently counter-intuitive results given in Figures 3 and 5. In L-series material, the presence of cement bridges between aggregates results in strong interaggregate bonding and so high values of UCS. By existing as distinct regions, however, cement hydration rates are reduced due to the lower exposed surface area, resulting in low hydration water quantities at 28 days. Suction conditions during cement hydration are difficult to comment on, given the lack of retention data (due to the complex interaction between water and cement and subsequent effects on temperature, humidity, salt content etc.), however the presence of large interaggregate voids within the material suggests that more free water might exist as menisci, rather than within filled pores, so that the proportion of free water available to contribute to material strength through suction is high (Beckett and Augarde, 2013). The development of the fine particle matrix with increasing compaction water content results in a less contiguous cement phase and

so a reduction in the contribution of interparticle bonding to UCS, but an increase in the rate of cement hydration due to the increased exposed surface area. The concurrent reduction in the number of large interparticle voids suggests that the contribution to material strength made by suction also reduces. At the highest compaction water contents, hydrated cement is fully held within the fine particle matrix so that its ability to contribute to interparticle bonding is at its lowest. By being spread throughout the fine matrix, however, the cement exposed surface area is at its largest so that this material displays the fastest cement hydration rates. Therefore, although they have the lowest compacted dry density, compaction water content and hydrated cement content, L-series specimens display higher values of UCS than both the O- and H-series specimens.

A transition from an aggregated structure to a matrix-dominated structure on increases in compaction water content, for a given compactive effort, has been observed by several authors for unstabilised clayey soils (Collins and McGown, 1974; Simms and Yanful, 2001; Monroy et al., 2010). This therefore suggests that the role of cement, at least during compaction, is also to control aggregation behaviour in the crushed limestone soil used here. It is therefore likely that a change in the initial cement content would result in a significant change in the nature of the material structures shown in Figures 6 to 8. It is not possible to comment on the nature of this change, given the evidence provided in this investigation, however it is a suggested topic for further study. Interpretations of results presented here must, therefore, be limited to similar materials and stabilisation regimes. These results have, however, demonstrated the key importance of the role of material structure and, hence, compaction regime, when determining the effectiveness of stabilisation regimes on material strengths.

4. Conclusions

This paper has presented the results of an experimental programme aimed at understanding the effects of compaction water content on the unconfined compressive strength of three series (L, O and H) of crushed limestone specimens, stabilised with 5% Portland cement.

Results of UCS testing suggested that lower compaction water contents produced higher material strengths, with L-series material achieving significantly higher values of UCS, despite being manufactured to the lowest value of ρ_d , than the same material compacted at or above the optimum value, for all tested curing times.

Freeze drying was used to arrest cement hydration after different curing periods in order to inves-

250 tigate the amount of hydrated cement, as interpreted by the amount of water lost to hydration, present
251 in specimens of each series. The seemingly counter-intuitive result was found that, despite their lower
252 strengths, specimens prepared to higher compaction water contents had a greater amount of hydrated
253 cement at the end of curing.

254 SEM analysis of material fragments showed that L-series material was characterised by isolated
255 particles or aggregates, bonded by cement bridges, and large interaggregate pores. Increases in com-
256 paction water content resulted in the formation of a fine particle matrix surrounding the larger parti-
257 cles/aggregates. The number of large interaggregate pores and cement bridges concurrently reduced,
258 with cement instead residing either on the surface of or within the fine matrix. At the highest com-
259 paction water content, cement bridges and interaggregate pores had disappeared entirely. It was argued
260 that the formation of this fine particle matrix was responsible both for the reduction in material UCS,
261 due to a reducing effectiveness of cement bonding, and the increase in hydrated cement contents, due to
262 greater exposed surface areas, with increasing compaction water content. It was observed that the for-
263 mation of a fine particle matrix, with a corresponding reduction in the volume of interaggregate pores,
264 with increasing compaction water content has previously been identified by several authors for unsta-
265 bilised clayey soils, suggesting that the role of cement in the material tested in this investigation was
266 similar to that of clay in those materials. Therefore, it is likely that the use of different Portland cement
267 contents will result in a different relationship between UCS, compaction water content and material
268 structure than that discussed here; this is a subject for future testing. Nevertheless, results presented in
269 this investigation have demonstrated the key role of material structure and initial water content as well
270 as the stabilisation regime when interpreting the strengths of stabilised materials.

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Table 1. Data for freeze drying tests. *Initial Water Content; **Final Water Content; ***Hydration Water; [†] Error on testing

Series	Curing time (days)	IWC* (g)	FWC** (g)	HW*** (g)
L	1	122	112	10
L	3	122	110	12
L	5	121	107	15
L	7	122	105	17
L	14	122	105	17
L	28	122	105	17
O	1	147	136	10
O	3	147	135	12
O	5	147	132	14
O	7	147	131	16
O	14	147	128	19
O	29	147	123	24
H	1	168	157	12
H	3	169	153	16
H	5	168	151	18
H	7	169	- [†]	- [†]
H	14	169	149	20
H	29	169	143	26

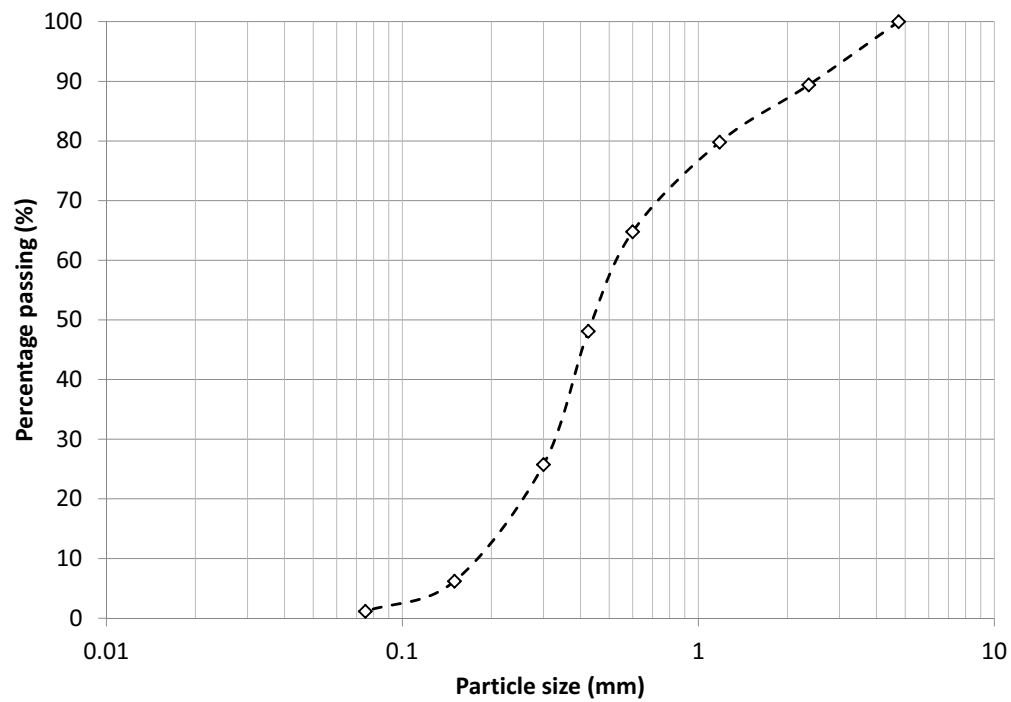


Fig. 1. Crushed limestone particle size distribution

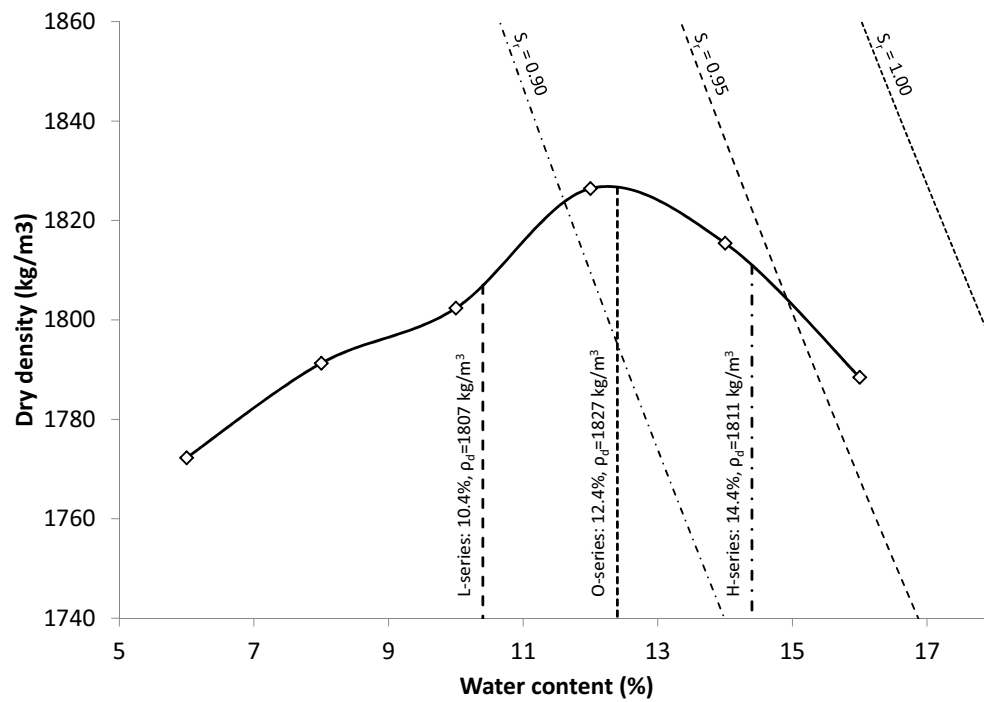


Fig. 2. Optimum water content testing ◇ and dry density values corresponding to -2% WC (L-series), OWC (O-series) and +2% WC (H-series). Lines for constant degrees of saturation (S_r) of 1.0, 0.95 and 0.9 are also shown.

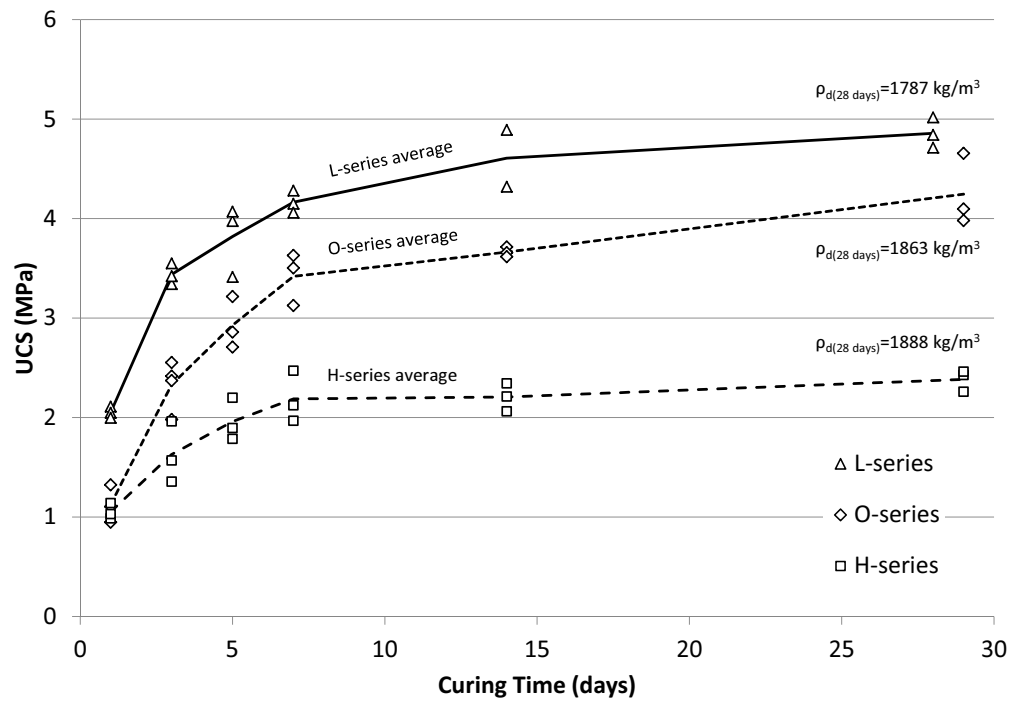


Fig. 3. Unconfined compressive strength results for the three series vs. curing time. Dashed lines represents the trend of the average UCS with curing time.

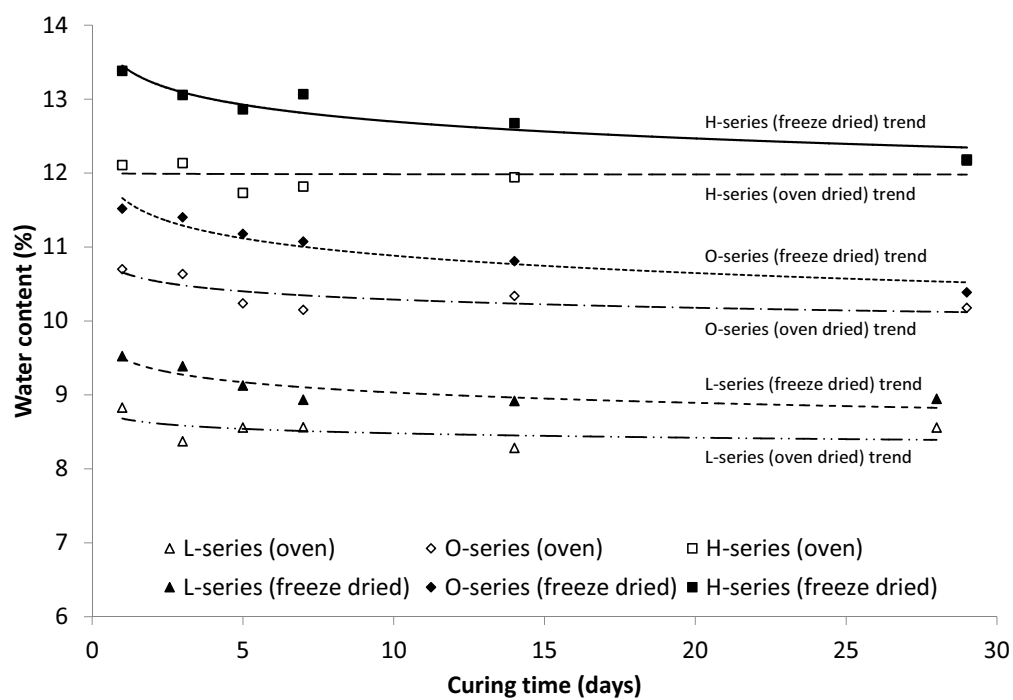


Fig. 4. Measurements of water content through time obtained via oven and freeze drying techniques.

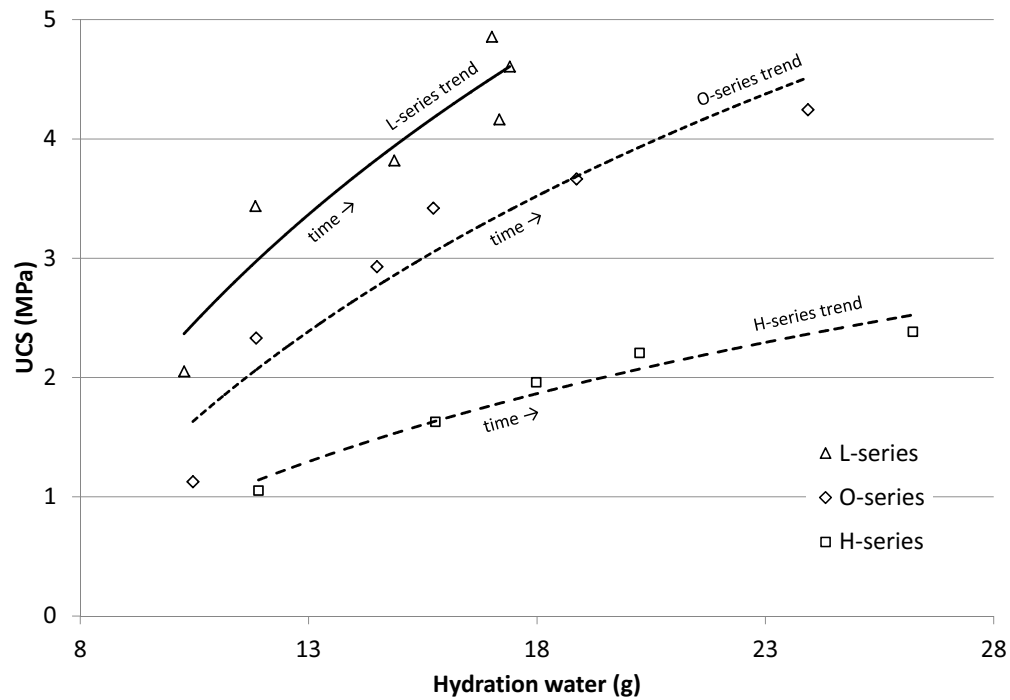


Fig. 5. Series unconfined compressive strength as compared to amount of water used in the cement hydration process.

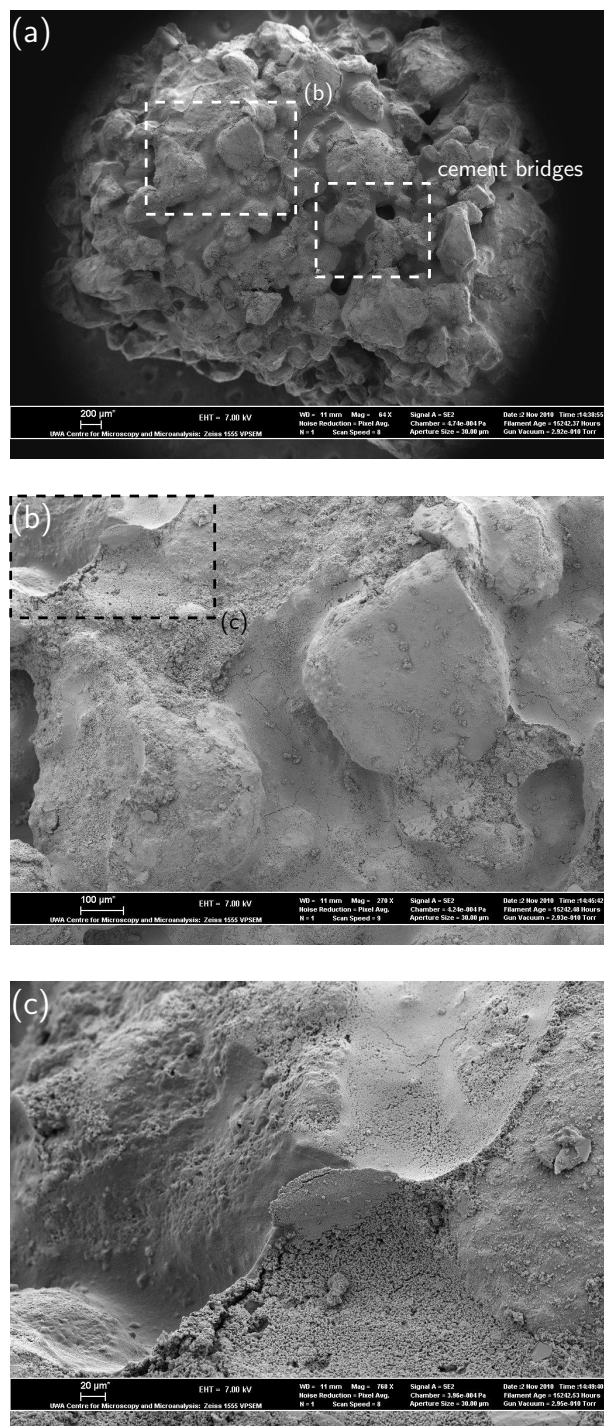


Fig. 6. SEM micrographs for L-series material fragments. Marked regions indicate positions of zoomed images.

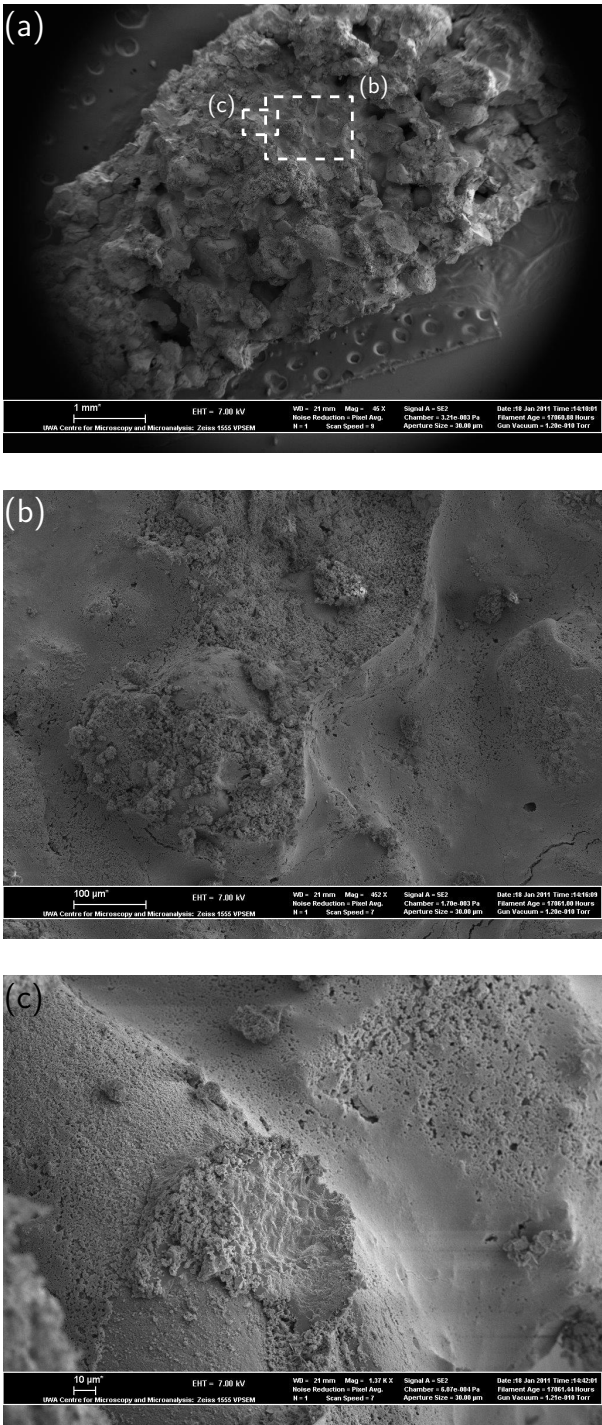


Fig. 7. SEM micrographs for O-series material fragments. Marked regions indicate positions of zoomed images.

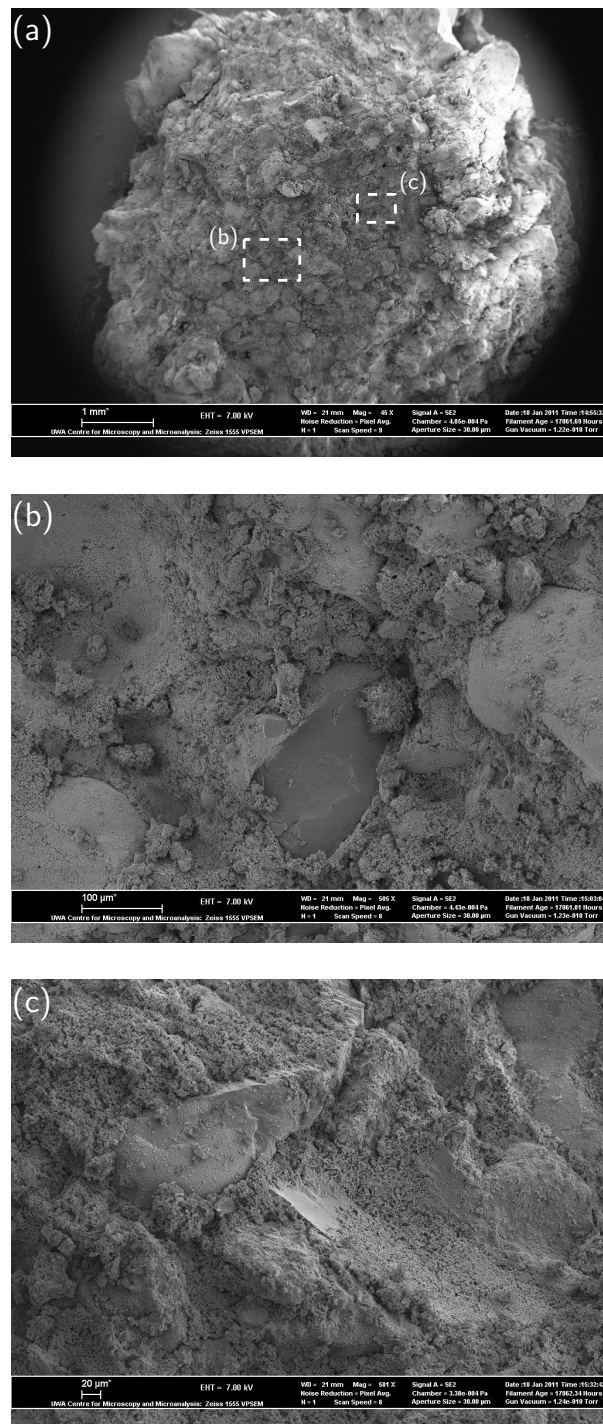


Fig. 8. SEM micrographs for H-series material fragments. Marked regions indicate positions of zoomed images.